MICROWAVE ASSISTED SYNTHESIS AND CHARACTERIZATION OF LSMO: COBALT FERRITE COMPOSITES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology
In
Ceramic Engineering

By Purab Das
Roll No- 108CR009

Department of Ceramic Engineering
National Institute of Technology
Rourkela
2012

MICROWAVE ASSISTED SYNTHESIS AND CHARACTERIZATION OF LSMO: COBALT FERRITE COMPOSITES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology
In
Ceramic Engineering

By Purab Das
Roll No- 108CR009

Under the Guidance of
Prof. Bibhuti B. Nayak

Department of Ceramic Engineering
National Institute of Technology
Rourkela

2012

National Institute of Technology
Rourkela

CERTIFICATE

This is to certify that the thesis entitled, "*Microwave assisted synthesis and characterization fo LSMO: Cobalt ferrite composites*", submitted by Mr. Purab Das (Roll no. 108CR009)in partial fulfillment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university / institute for the award of any Degree or Diploma.

Prof. Bibhuti B. Nayak

Associate Professor

Department of Ceramic Engineering

National Institute of Technology

Rourkela- 769 008

ACKNOWLEDGEMENTS

With deep regards and profound respect, I avail this opportunity to express my deep sense of gratitude and indebtedness to Prof. Bibhuti B. Nayak, Department of Ceramic Engineering, NIT Rourkela, for introducing the present research topic and for his inspiring guidance, constructive criticism and valuable suggestion throughout in this research work. It would have not been possible for me to bring out this thesis without their help and constant encouragement. I would also like to thank Prof. S. K. Pratihar, Department of Ceramics Engineering, NIT Rourkela, for helping and carrying out different characterization**.** Special thanks to Subrat Mohanty, Geetanjali Parida, as a PhD Scholars of the department for being so supportive and helpful in every possible way. I am highly grateful to all staff members of Ceramic Engineering, N.I.T., Rourkela, for their help during the execution of experiments.

May, 2012

Purab Das

108CR009

Contents

LIST OF FIGURES

LIST OF TABLES

Abstract

Manganite-Ferrite Nano composite materials are of great technical applicability, as these materials can be used as read heads for hard disks, magnetic storage and sensing devices.These composites are having an inherent property of magnetic coupling between these two phases affecting the microstructural and magnetic properties. The main objective of this work is to study the effect of CF addition on the microstructural and magnetic properties of LSMO: CF composites using microwave oven. In this work, microwave assisted synthesis was carried out for the preparation of (1-x) mol % $La_{0.67}Sr_{0.33}MnO_3$ (LSMO): x mol % CoFe2O4 (CF) (where x= 2, 5, 10,and 15) powder composites by using microwave oven. Different characterization techniques such as XRD, SEM, density and M-H loop have been performed to study the properties of these composites.Minimum calcinations temperature for pure LSMO and CF powders is 800 °C as observed from thermal analysis. X-ray diffraction analysis confirms the presence of two phases with either LSMO or CF, without any impurities. LSMO and ferrite phases are uniformly distributed with nearly spherical morphology as observed from the SEM micrographs. The particle size of LSMO is smaller as compared to CF in these composites. SEM with EDAX confirms the two phase system of all LSMO: CF composites.

Keywords: Microwave-assisted synthesis; CMR-ferrite Nano composites; Coercivity; Microstructure.

Chapter-1

General Introduction

Relevance of manganite.

Relevance of ferrites.

Importance of manganite-ferrite Nano composite.

1.1 Relevance of manganite.

The perovskite manganites are of the general formula $RE_{(1-x)}A_xMnO_3$ (RE = trivalent rare earth element such as La, Pr etc. and A = divalent alkaline earth ions such as Ca, Sr, Ba etc.). Theyhave attracted growing attention due to the colossal magneto resistance (CMR) properties.In perovskite structure, (RE, A) element occupy the A-site position (corner of a cube) and Mn occupies the B-site position (body center of a cube). All the face centered positions are occupied by oxygen [1].

Depending on the composition, manganite shows a variety of magnetic and electric phenomena, including ferromagnetic, antiferromagnetic, charge, and orbital ordering [2]. If the site *A* is partially occupied by one divalent atom such as Ca or Sr, then Mn3+ and Mn4+ coexist in the samples and the compounds show different behavior as the temperature is changed. The perovskite manganites have generated considerable interest in recent years because of their colossal magneto resistance (CMR) behavior. The effectiveness of these materials is directly related to the percentage change of resistance in an external magnetic field. A negative MR was first found in perovskite manganites, exhibiting huge decrement in electrical resistivity in the presence of magnetic field. With increase of temperature, it increases up to a temperature, T_{MI} (called metal- insulator transition temperature) beyond which it decreases having a negative temperature coefficient of resistance while maintaining a large magnitude. This metal to insulator transition at this temperature TMI usually accompanied by ferromagnetic to paramagnetic transition. A composition of x~0.33 is ideal to observe a good metal-insulator transition and negative colossal magneto resistance (CMR) [3].

$La_{0.67}Sr_{0.33}MnO_3$ (LSMO) possesses difference of properties from $La_{0.67}Ca_{0.33}MnO_3$ (LCMO). In LSMO case, the A-site ion size is being larger. The ferromagnetic metallic regime in LSMO

extends over larger values of x. It also possesses the highest value of curie temperature (Tc = 370K) and combines low carrier density (10^{21} to10^{22} cm^{-3}) with a high spin polarization of charge carriers among CMR materials, which makes it the most promising in room temperature application [4].

1.2 Relevance of Ferrites

Ferriteshave the generic formula AB_2O_4, where "A" is a divalent ions such as Ni^{2+}, Co^{2+}, Mg^{2+}, Fe^{2+}, Mn^{2+}, $Zn^{2+}$and "B" is a trivalent ions such as Fe^{3+} and Al^{3+}. They are having spinel type structure.The structure of spinels is described as having an oxygen ion sub lattice arranged in a cubic close-packed arrangement with cations occupying various combinations of the octahedral (O) and tetrahedral (T) sites. The cubic unit cell is large, comprising 8 formula units and containing 32 O and 64 T sites. Spinels are divided into two categories such as normal and inverse spinel. In normal spinel, the divalent cations "A" are located on the tetrahedral (T) sites and the trivalent cations "B" on the octahedral (O) sites. Generally Zn, Cd etc. prefers normal spinels. $NiFe_2O_4$ (NF) or $CoFe_2O_4$ (CF) has an inverse spinel crystal structure. In the case of an inverse "A" cation occupies one half of the octahedral coordination sites and half the "B"cation occupies the other half "O" sites as well as all "T" sites. The magnetic properties of these ferrites arise largely from their structural features and the nature of the distribution of the ions in the sub lattices.In spinels, the interaction between A and B atoms is always almost antiferromagnetic (they have opposite spins) so they cancel one another and the rest of 4 Fe^{+3} ions are aligned in the same direction with field so they are responsible for net magnetization. In terms of magnetic properties, ferrites are often classified as "soft" and "hard" which refers to their low or high corecivity of their magnetism, respectively. Soft magnetic materials are used in devices (such as transformer core) that are subjected to alternating magnetic field and in which energy loses must

be low. Soft magnetic materials have high initial permeability and low corecivity (easily magnetized and demagnetized). Low value of corecivity, gives easy movement of domain walls but they are restricted by the structural defects like voids or any nonmagnetic phases, so soft magnetic materials are must be free of such structural defects. They have also high electrical resistivity. Hard magnetic materials are utilized in permanent magnets, which must have a high resistance to demagnetization. It has high remanence, corecivity, saturation flux density as well as low initial permeability and high hysteresis energy loss [5].

CF is a well-known hard magnetic material with high corecivity and moderate magnetization. These properties along with their great physical and chemical stability make CF suitable for magnetic recording applications such as audio and video tapes and high density digital recording disks.

1.3 Importance of manganite: ferrite Nano composites

Manganite-ferrite nanocomposites due to their magnetic coupling between the two phases are well known,which affects the microstructural and magnetic properties. Nano composites of manganite, a colossal magnetoresistance perovskite and ferrite an insulating magnetic oxide are of interest because of their unusual magnetic as well as electrical properties. The reason for studying such composites is that the presence of the insulating phase forces the electric current to meander through the CMR grains, thereby increasing significantly the contribution of the grain boundaries to the conduction process [6]. This leads, in turn, to an enhancement of electrical and magnetic properties. Due to both fundamental and significant technological importance, the research on manganite-ferrite Nano composites has been very actively developed in recent years.

Chapter -2

Literature Survey

Manganite: ferrite composite Synthesis techniques.

Advantages of microwave-assisted synthesis route.

Manganite: ferrite Nano compositesynthesis by kitchenMicrowave-oven.

Literature reviewSummary.

2.1 Manganite: ferrite composite Synthesis techniques

There are different methods been used for enhancing these electrical and magnetic properties such as: substitution doping, grain size reduction, distribution of the manganite grains in a non-magnetic insulating matrix and magnetic insulating matrix. LSMO: $SrFe_{12}O_9$ Nano composite system [7] by chemical co-precipitation method. The composites of LSMO: SFO are also prepared using solid-state sintering. In which, it was possible to enhance spin disorders at the grain boundaries and interfaces of LSMO grains by introducing a second magnetic material into LSMO grains in order to enhance electrical and magnetic properties.

Magnetic nanoparticles, besides its important technical applications in magnetic refrigerators, magnetic recording, magnetic fluids, and biomedicine, it mightalso be applied for microwave absorption at high frequency over gigahertz, ascribed to the high Snoek's limit.

Xiong et al. [8] have studied $La_{0.7}Ca_{0.2}Sr_{0.1}MnO_3$ (LCSMO)/$CoFe_2O_4$ (CFO) composites, which were prepared by a standard ceramic technique. Compared with pure LCSMO, the MR effect of the composites is enhanced in low magnetic fields of 3 kOe at a wide temperature range below Tc. The enhanced MR at low field (LFMR) is related to the enhancement of spin-dependent tunneling of electrons and spin-dependent scattering at the surfaces between LCSMO grains.

Yan *et al.* [9] have investigated the LFMR of the LSMO/CF composite for a single composition of 20 wt.% CF. The resistivity of the composite is about an order of magnitude larger than that of the same grain-sized pure LSMO. A large LFMR has been obtained in this composite compared to pure LSMO. Since the spin-dependent scattering of the conduction electrons at the grain boundaries is highly field sensitive, the magnetic scattering of the polarized charge carriers may be responsible for the electrical and magnetic properties.

2.2. Advantages of microwave-assisted synthesis route

Microwaves are electromagnetic waves with wavelengths from 1 mm to 1 m and corresponding frequencies between 300 MHz and 300 GHz. 0·915 GHz and 2·45 GHz frequencies are commonly used for microwave heating [10]. These frequencies are chosen for the microwave heating based on two reasons. The first is that they are in one of the industrial, scientific and medical (ISM) radio bands set aside for non-communication purposes. The second is that the penetration depth of the microwaves is greater for these low frequencies.

In microwave heating, unlike conventional heating, heat is generated internally within the material instead of originating from external sources. As a result of internal and volumetric heating, thermal gradients and direction of heat flow in microwave heated materials can be just the opposite of those in conventional methods**.** It offers a clean, cheap and convenient method of heating often resulting in higher yields and shorter reaction times. Applications of microwave processing are indeed very wide. For instance, it can include processing of solution and suspension, drying, organic material burnout, clinkering, sintering of ceramics and ceramic composites, preparation of specialty ceramics, plasma processing, processing of polymers and polymer composites, fabrication of functionally graded materials, joining, fiber drawing, melting, reaction synthesis of ceramics and a host of very promising, new, advanced applications.

David et al. [11] investigates about the microwave synthesis which has been modified so thatsyntheses involving the refluxing of organic solvents can be safely and conveniently under taken. There was a misconception between researchers that all metals reflect microwave or cause plasma formation, and hence cannot be heated, except exhibiting surface heating due to limited penetration of the microwave radiation. The researchers did not notice that this relation is valid

only for sintered or bulk metals at room temperature, and not for powdered metals and/or at higher temperatures. Now it has been found that the microwave sintering can also be applied as efficiently and effectively to powdered metals as to many ceramics.Microwave processing has gained worldwide acceptance as a novel method for heating and sintering a variety of materials, as it offers many advantages in terms of enhanced diffusion processes, reduced energy consumption and processing cost, very rapid heating rates and significantly reduced processing times, decreased sintering temperatures, improved physical and mechanical properties, simplicity, unique properties, new materials and products and lower environmental hazards, which are not observed in conventional processes.

2.3. Manganite: ferrite Nano composite synthesis by kitchen Microwave-oven

There are different synthesis techniques that are used to prepare Nano composite materials. Among all the synthesis techniques, microwave synthesis technique is now well documented. This technique is highly versatile and can be used for the synthesis of a wide variety of materials. This technique has the inherent advantage of uniformly distributing the two phases and the two phases will also have a similar grain size distribution as they will be subjected to identical processing conditions. This will overcome the limitations of asymmetric grain sizes for the two phases encountered and thus facilitate study of possible electronic interactions between the two phases, LSMO and CF. The interesting aspect of these Nano composites is that above the transition temperatures corresponding to LSMO phase, the Nano composite will be electrically insulating but magnetic due to the presence of CF. Below the transition temperatures of LSMO the composite will be conducting and also magnetic with a CMR behavior. This temperature dependent behavior makes it attractive for innovative applications in magnetic information

storage. The LSMO phase however loses its negative magnetoresistance behavior due to a substitution of Mn with either or both Co and Fe. Nayak et al. [12] have studied the properties of nano grained LCMO: NF ($NiFe_2O_4$) composites which are prepared in situ microwave refluxing technique. This technique has the inherent advantage of uniformly distributing the two phases and the two phases will also have a similar grain size distribution as they will be subjected to identical processing conditions. This will overcome the limitations of asymmetric grain sizes for the two phases encountered and thus facilitate study of possible electronic interactions between the two phases, LCMO and NF.

2.4. Summary of literature review

From literature it was found that:

- Doping in A or B site, creates a high density of disordered areas such as grain boundaries and distribution of the perovskite manganite in different matrices which have been used as an attractive approaches to enhance the electrical and magnetic properties of manganite-ferrite Nano composites.
- Microwave refluxing is a novel technique to synthesize manganite-ferrite composites at a nano level scale for different applications.
- There are few literatures on the effect of ferrite addition on the electrical and magnetic properties of manganite by different synthesis routes.

In this work, finally microwave assisted synthesis was carried out for the preparation of $La_{0.67}Sr_{0.33}MnO_3$ (LSMO):$CoFe_2O_4$ (CF), Nano composites using kitchen microwave oven. Different compositions of (1-x) mole% LSMO: x mol% CF composites (where x= 2,5,10, and 15) have been synthesized using kitchen microwave oven. Different characterizations techniques such as XRD, SEM with EDAXand M-H loop test have been performed and the results are discussed in detail.

Chapter – 3

Experimental Analysis

Manganite: ferrite Nano composite synthesis by kitchen Microwave-

ovenCharacterization techniques for the analysis of composites

X-ray diffraction (XRD)

Scanning electron microscope (SEM)

Magnetization

Density

3.1. Manganite: ferrite Nano composite synthesis by kitchen Microwave-oven

The preparation of pure LSMO powder includesthe mixing of stoichiometric equivalents of La-acetate, Sr- chloride and Mn-acetate with ethylene glycol and this mixture is used as a precursor I. For preparation of pure CF powder, stoichiometric equivalents of cobalt chloride and Iron chloride were mixed with ethylene glycol and this mixture is used as a precursor II. For the composite case (composition mention in objective section of chapter 2) i.e. precursor III is the mixture of precursor I and II.

The individual precursor solution was kept at 80 °C under constant stirring condition with the help of a magnetic stirrer. It is observed that all the particles of the solution been generated are not completely mixing within the solution even though there is continuous heating and stirring condition. Therefore the pH of this acidic precursor solution was increased by addition of KOH solution.The initially turbid solution now becomes clear with the addition of this KOH solution and form a gelly like structure at pH = 11. These gel precursors were taken for further processing by microwave refluxing. A commercial microwave generator operating at 2.5 GHz and 980 Watts was used to heat the precursor solution to an average of 473 K, boiling point of ethylene glycol. This was later condensed by refluxing with circulating water and recycled. The solution was subjected to microwave heating and refluxing for a period of 1hour. Now the precipitate obtained at the end of this experimental set up was washed thoroughly with distilled water, dried and calcined at 800 °C for 1h. The calcined powder was pelletized and sintering was done at 1200 °C for 2h.Structural, microstructural and magnetic properties of these annealed samples have been studied and analyzed.

3.2 GENERAL CHARACTERIZATION

3.2.1. X-ray diffraction

Phase analysis by X-ray diffraction (XRD): Phase analysis was carried out of the synthesized calcined manganite–ferrite composite (LSMO-CF) powder using room temperature powder X-ray diffraction with filtered 0.154056 nm Cu Kα radiation. Samples are scanned in a continuous mode from 20° – 80° with a scanning rate of 0.04 (°) / 1 (s). The phase purity and the crystallinity of the composite material powder were aimed to be achieved.

3.2.2. Scanning Electron Microscope:

Microstructural features along with chemical compositions (using EDAX) were studied using Scanning Electron Microscope (JSM 6480 LV JEOL, Japan). Through which the chemical purity was analyzed and even the distinction between different phases were analyzed. With the help of EDAX mechanism also the percentage of different individual phases were measured.

3.2.3. Magnetization

Pulse field hysteresis loop tracer (MAGNETA) was used to trace MH loop for sintered pellets. M-H loop can measure up to a magnetic field of 0.5 Tesla. The machine was first calibrated using standard sample having saturation magnetization of 72.4emu/g. Here the calcined pallet was used to determine the characteristic behavior of magnetization for different composition of manganite-ferrite composite (LSMO-CF) pallets. The graph also gives idea about the highest magnetization that can be achieved with different composition of manganite-ferrite composite and also about the stability or coercivity value for different compositions. Hence is a very important characterization technique for the determination of scope of application in magnetic tip heads for the different composition of composites.

3.2.4. Density

Density was measured by using vacuum assisted soaking of pellets in kerosene medium for about 2hr. The dry weight, suspended weight, and soaked weight were taken to calculate bulk density using the following formula.

Bulk Density =dry weight/(soaked weight-suspended weight) × density of liquid medium

Liquid medium in our case is kerosene with density of 0.80 g/cm3. Where, D, W, S stands for dry weight, soaked weight and suspended weight respectively of the sample. Similarly the apparent porosity of the sintered pallet was determined giving an idea regarding the overall density of the composite structured manganite-ferrite composite(LSMO-CF).

Similarly the apparent porosity is measured through the formulae:

Apparent porosity =(soaked weight – dry weight)/ (soaked weight – suspended weight). In case of the apparent porosity there is no need of multiplying the density of the medium to the given formulae as it does not affect the stability of the system.

Chapter – 4

Results & Discussion

Analysis of microwave-assisted synthesized LSMO-CF Composite

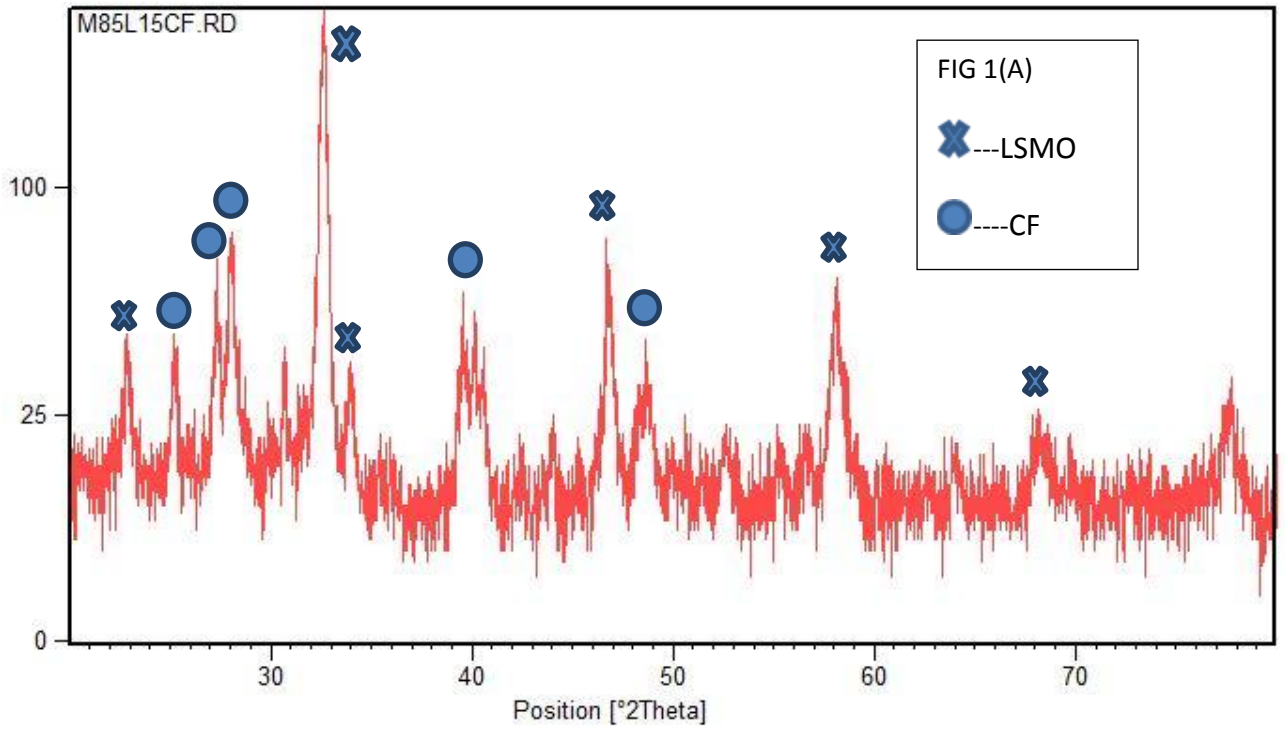
Characterization Techniques (DSC-TG, XRD, SEM, Density, Magnetization).

4.1 Analysis of microwave-assisted synthesized LSMO -CF composites.

In this section, pure phase of LSMO and CF have been synthesized by microwave refluxing process. The detail experimental procedure was described in Chapter 3, section 3.1 phase purity, particle morphology; density and magnetization have been studied using XRD, SEM, Archimedes principle and M-H loop, respectively. In this section structural, microstructural and magnetic properties of pure phase LSMO and CF powders were discussed in detail. Further we describe the synthesis of (1-x) mole % LSMO: x mole % CF (where x = 2,5,10 and 15) composite by using microwave refluxing technique. The detailed experimental procedure is explained in chapter 3, section 3.1. Structural, microstructural, density and magnetic properties are described in detail.

4.1.1 Structure

XRD patterns of sintered LSMO: CF composites with different molar percentage are shown in Fig.1 (A)-(D). All the peaks are identified with either LSMO (orthorhombic) or CF (cubic) as mentioned in the XRD patterns with the help of JCPDS (file no: 72-0841 for LSMO and 22-1086 for CF). No secondary phase has been detected, indicating that interfacial reactions between LSMO and CF grains are negligible. With increasing CF percentage, the intensity of (100) plane (2 theta = 32.23) reflection from the LSMO phase decreases, whereas the intensity of (311) reflection from the CF phase gradually increases.


M85L15CF.RD
FIG 1(A)
---LSMO
----CF
100
25
0
30
40
50
60
70
Position [°2Theta]

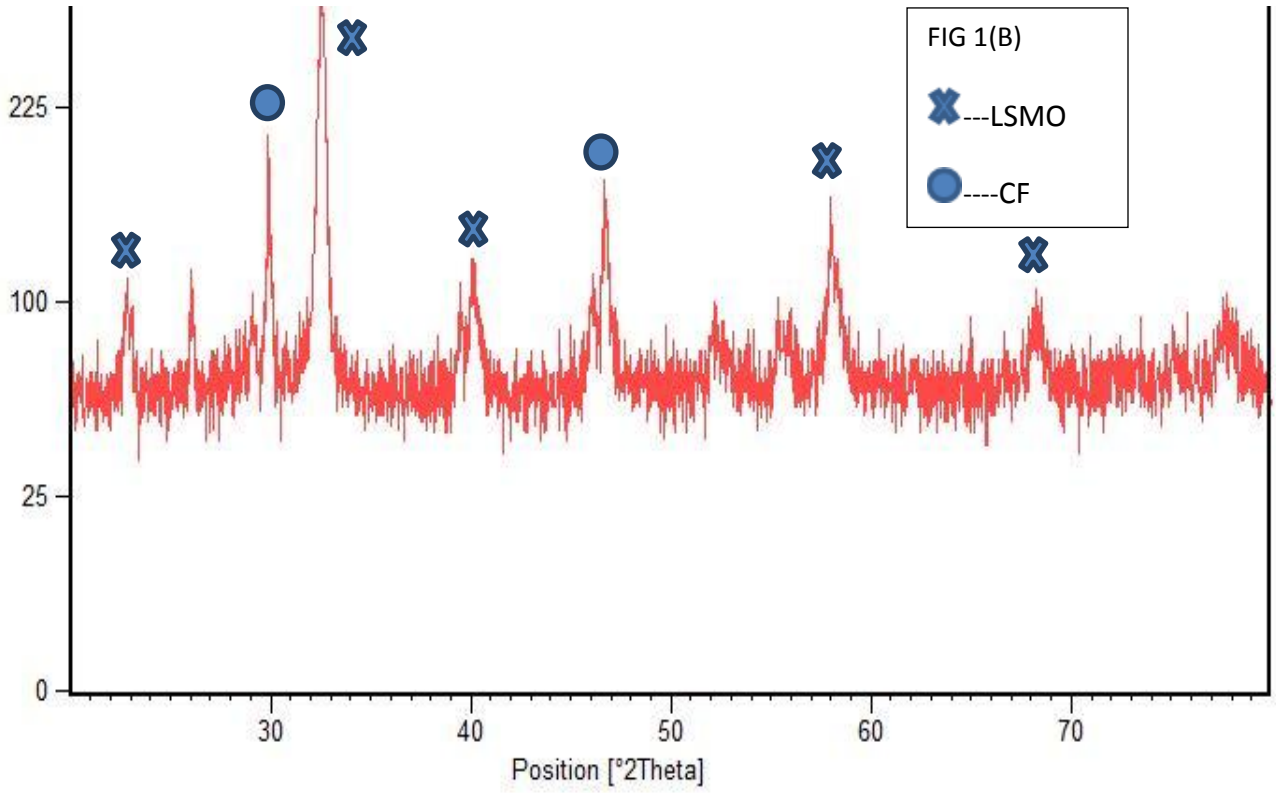


FIG 1(B)
---LSMO
----CF
225
100
25
0
30
40
50
60
70
Position [°2Theta]

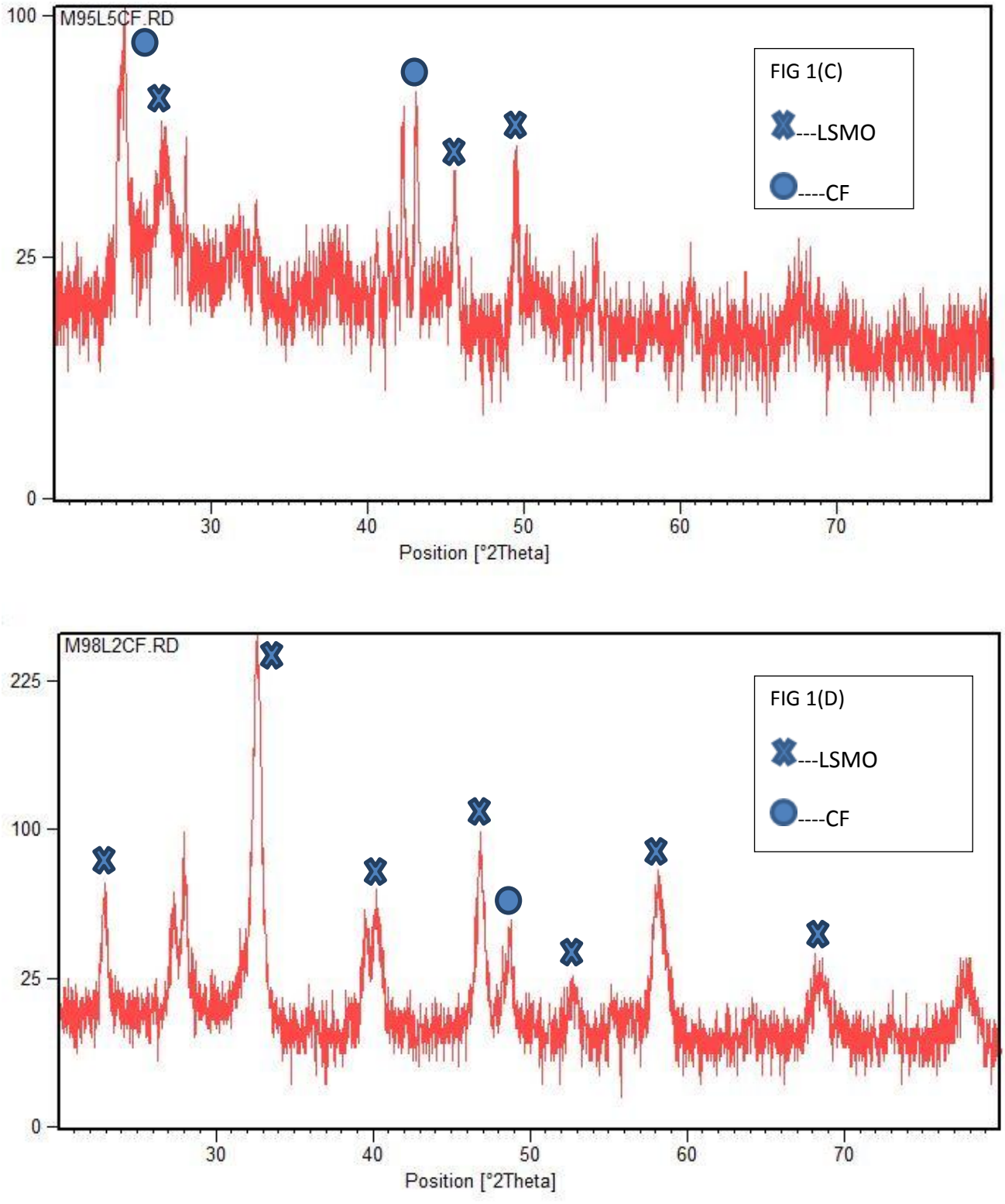


Fig.1 XRD patterns of (A) 85LSMO:15CF (B) 90LSMO:10CF (C) 95LSMO:05CF (D) 98LSMO:02CF powders.

4.2.2 Microstructure

Phase distribution and morphology were performed using back-scattered mode SEM on the sintered LSMO: CF composites. Fig.2 (a), (b), (c) and (d) shows the SEM micrographs along with EDAX as well as number of grains as a function of grain size (from SEM micrographs) for the composites 85 mole% LSMO: 15 mole% CF, 90 mole% LSMO: 10 mole% CF, 95 mole% LSMO: 05mole% CF, and 98 mole% LSMO:02 mole% respectively.The LSMO: 02 mole % CF and 05 mole % CF have similar microstructure (irregular multi-facet shape), whereas LSMO: 15 mole% CF composites have different morphology (nearly spherical) as seen from the SEM micrographs. From SEM images as well as EDAX analysis, it was confirmed that the white portions indicates the presence of LSMO and the black portion indicates the presence of CF. From microstructural analysis, it was also confirmed that the number of grains are in the range between 200 nm to 500 nm for all composites. Maximum number of grains is in the range of 200 nm for LSMO and for CF, the range is above 500 nm.

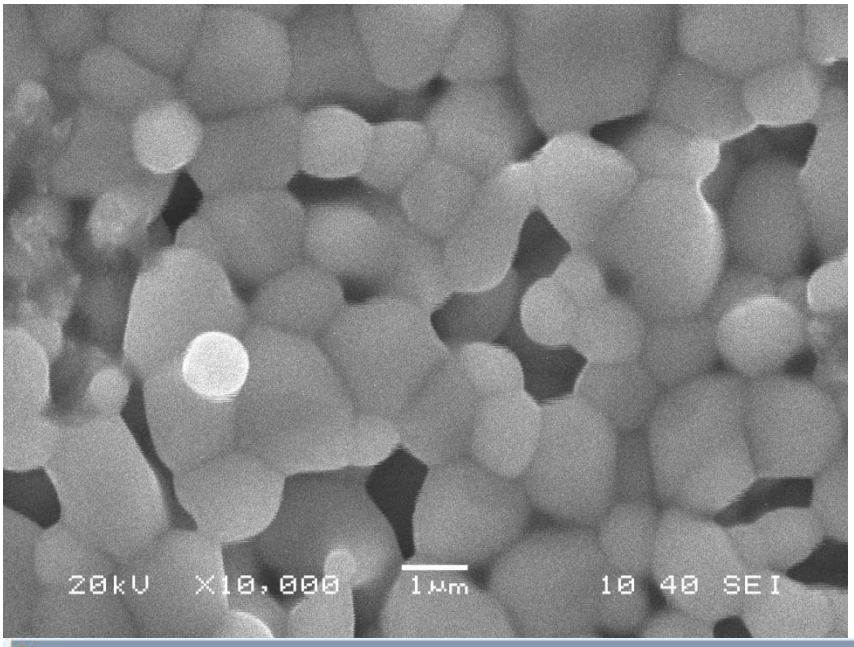


Fig. 2.a

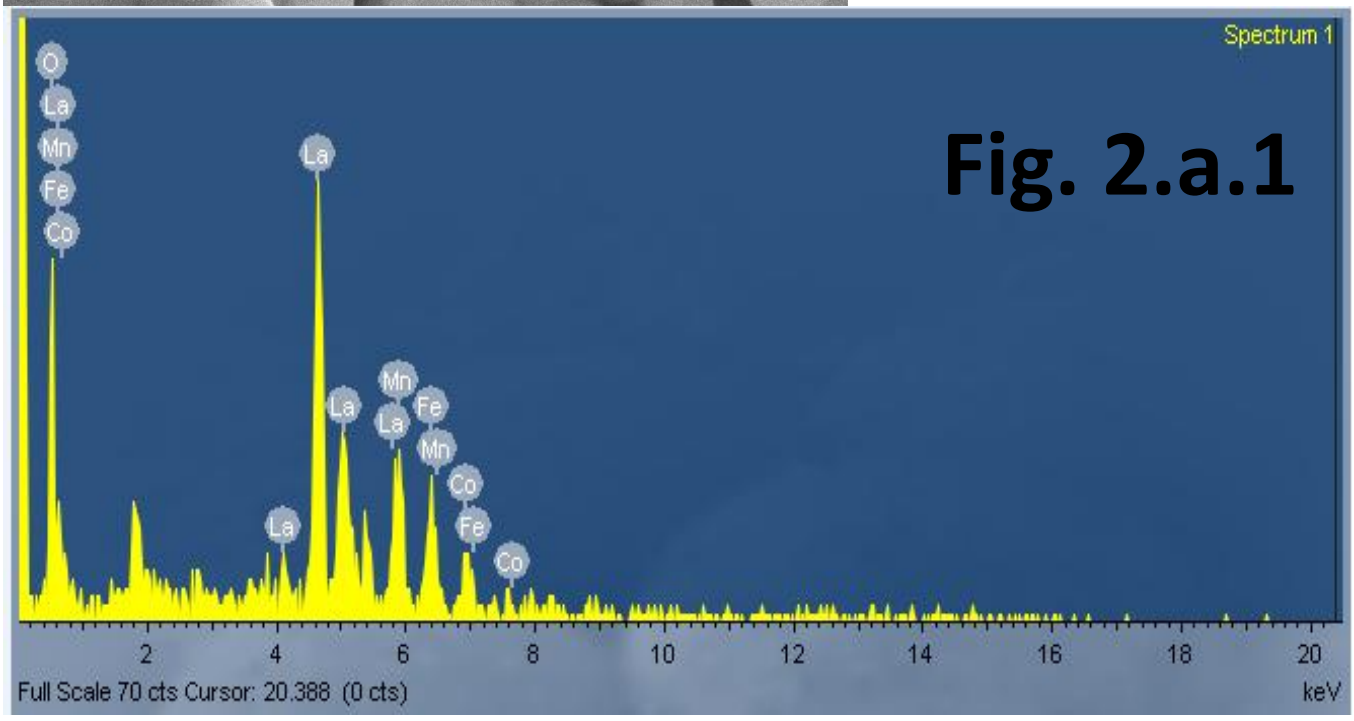


Fig. 2.a.1

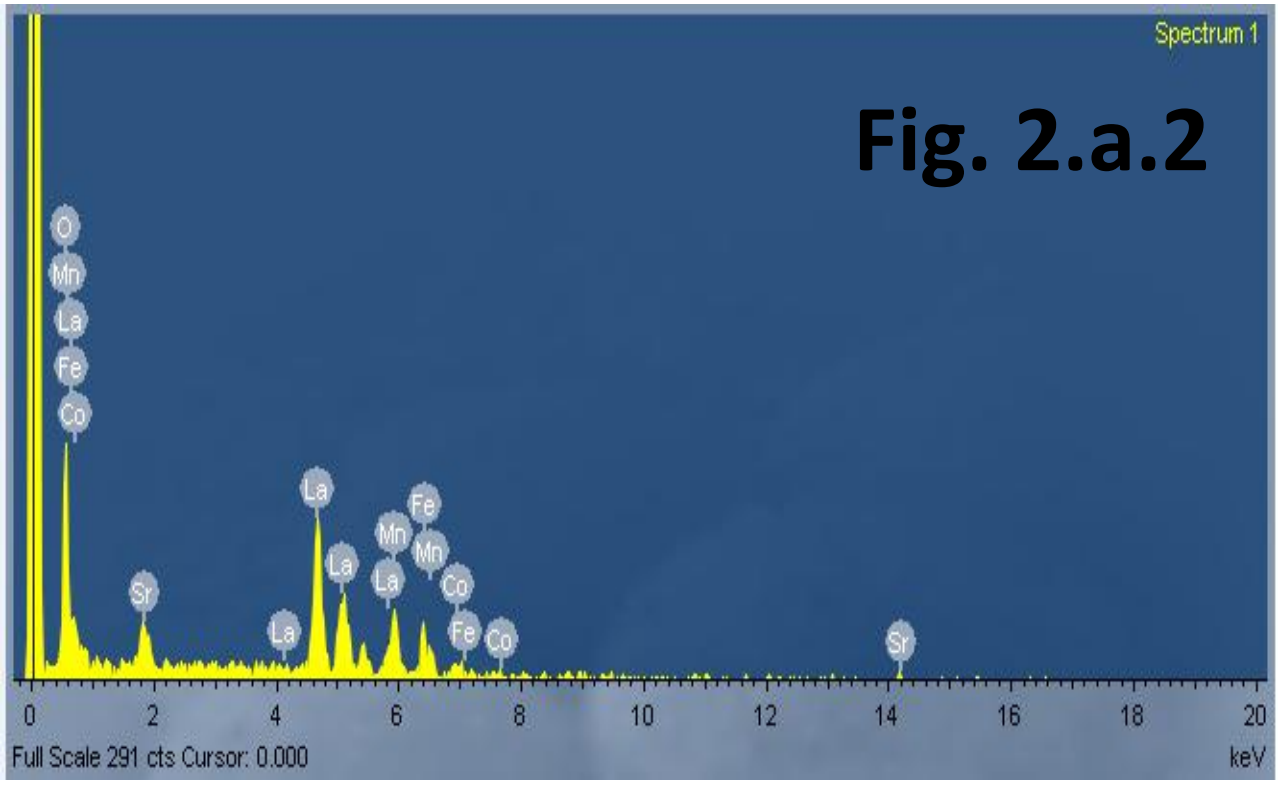


Fig. 2.a. SEM Graph and EDAX for the composite composition 85LSMO:15CF

Fig. 2.a.1. This represents the EDAX for the CF nanoparticle.

Fig. 2.a.2. This represents the EDAX for the LSMO nanoparticle.

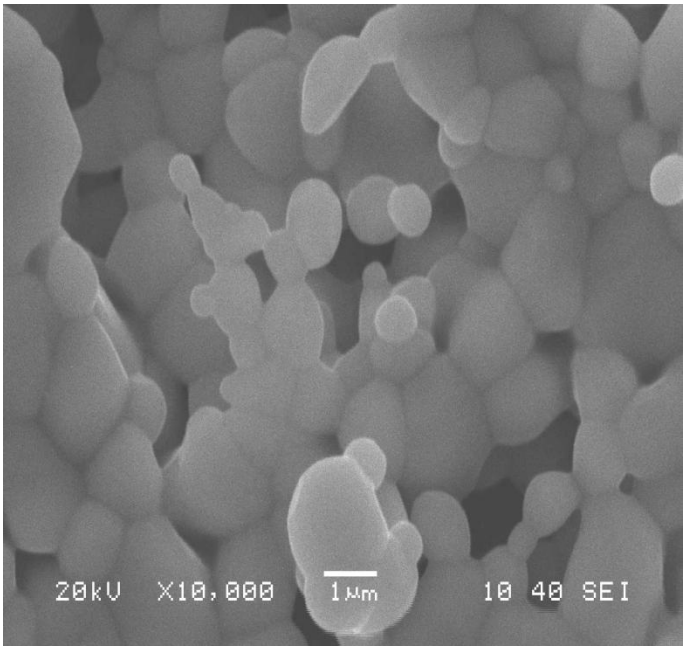
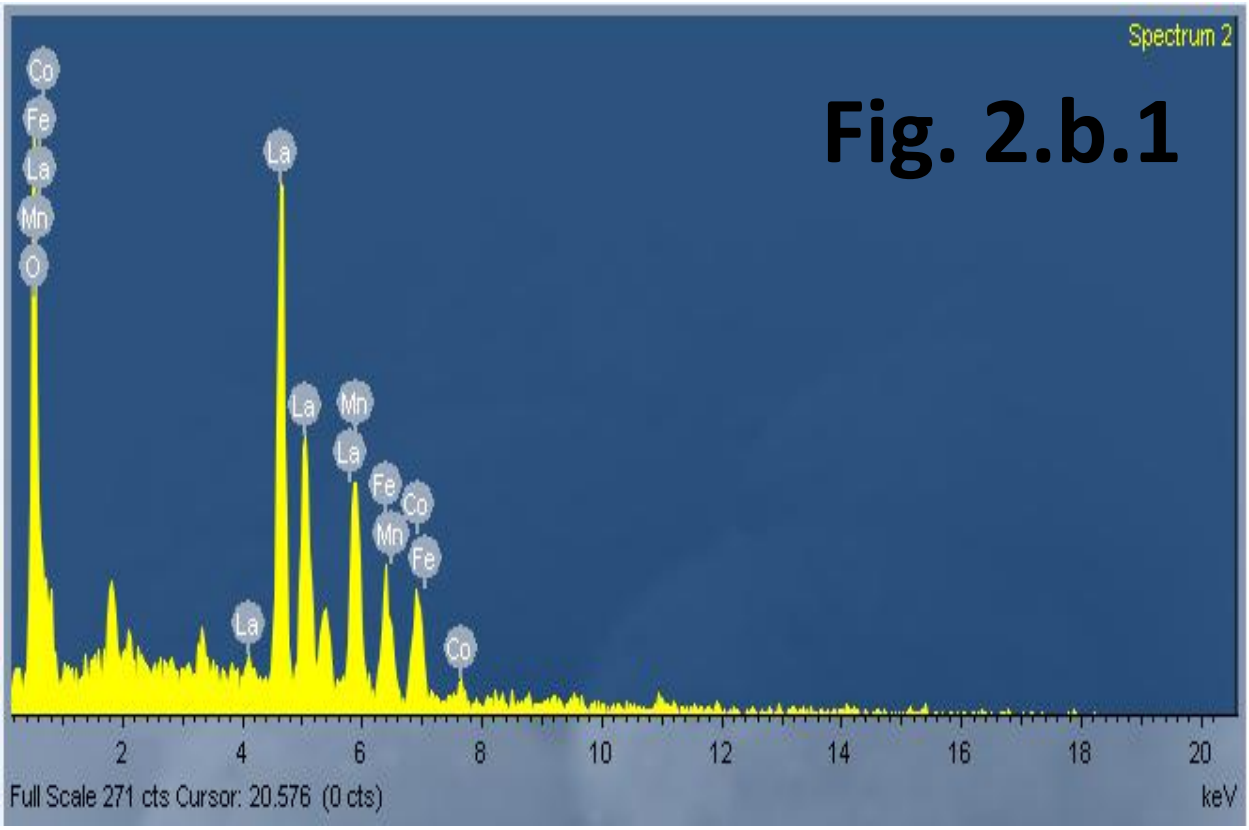


Fig. 2.b



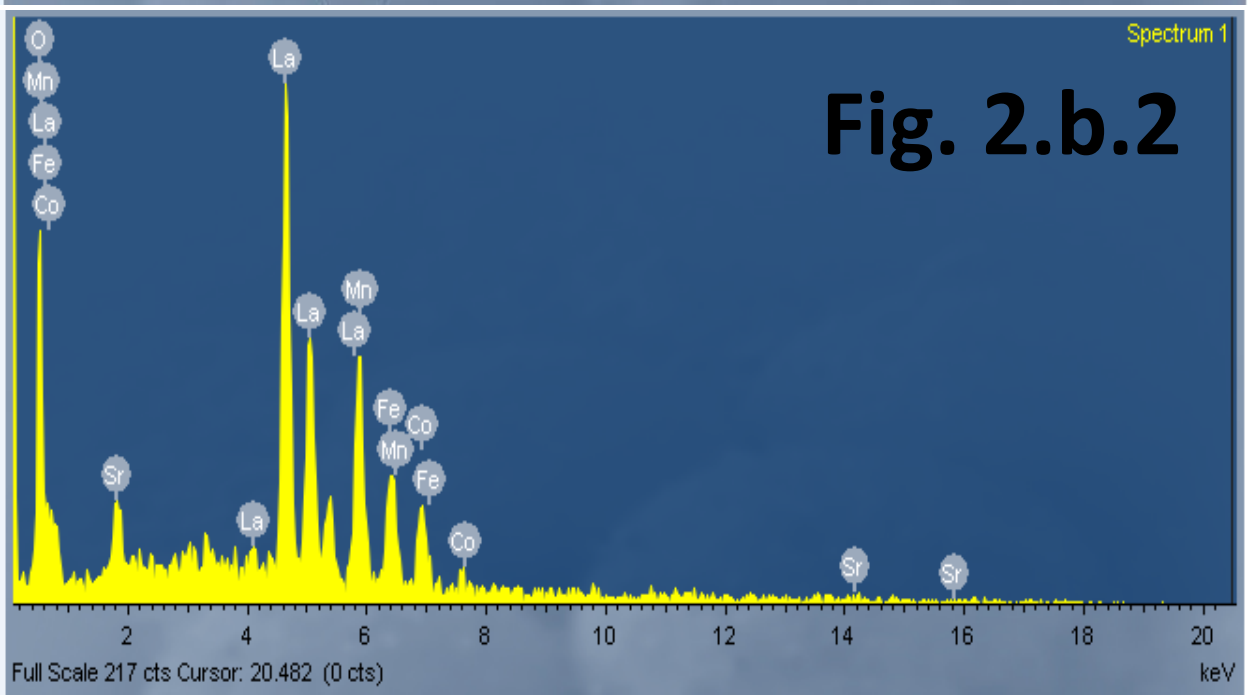


Fig.2.b SEM Graph and EDAX for the composite composition 90LSMO:10CF
Fig.2.b.1 This represent the EDAX for the CF nanoparticle
Fig.2.b.2 This represent the EDAX for the LSMO nanoparticle

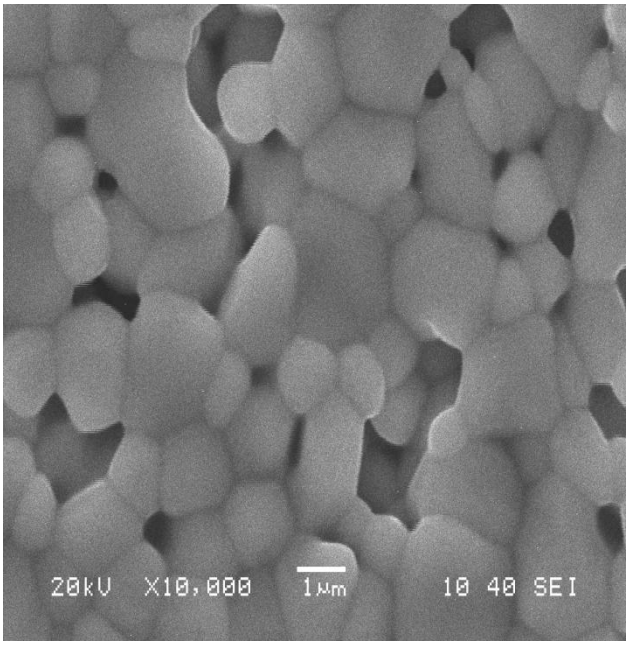


Fig. 2.c

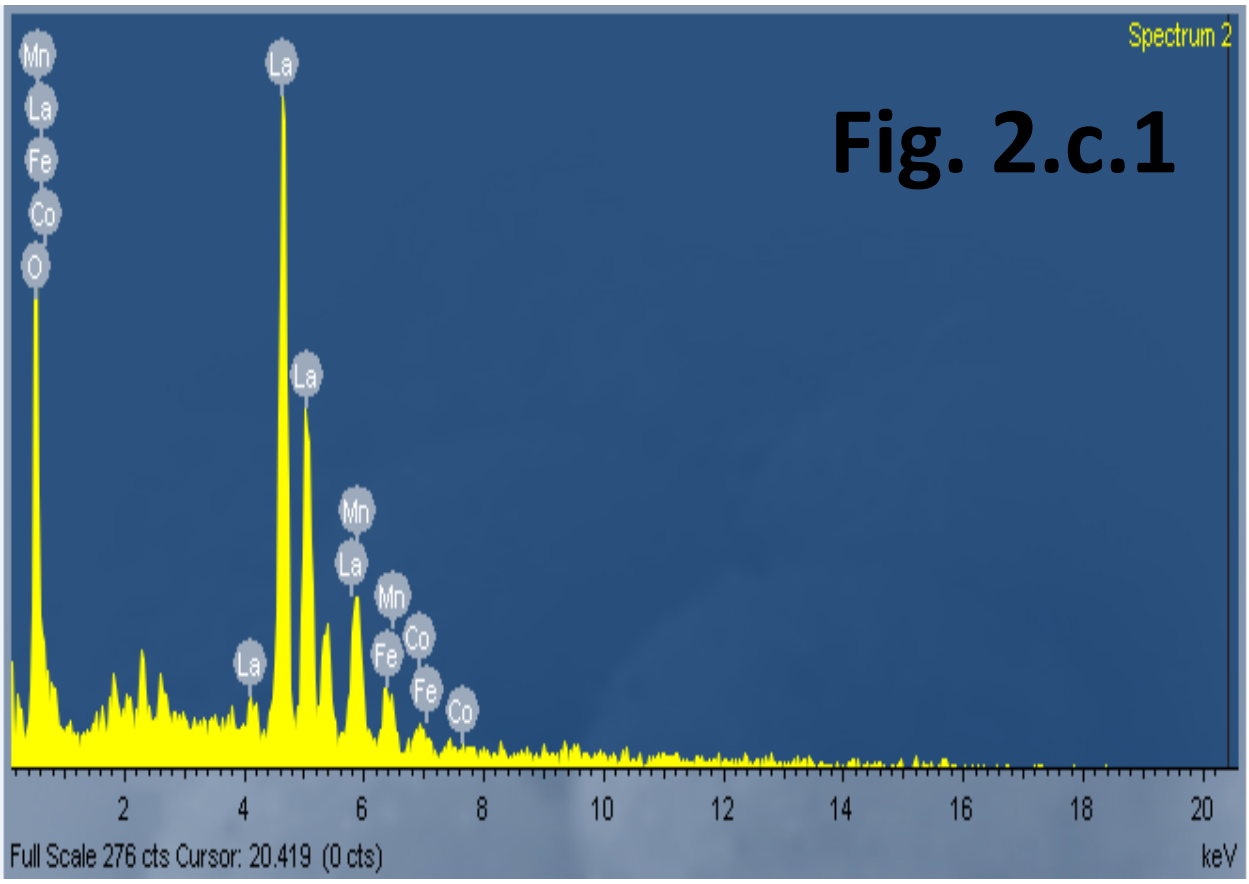


Fig. 2.c.1

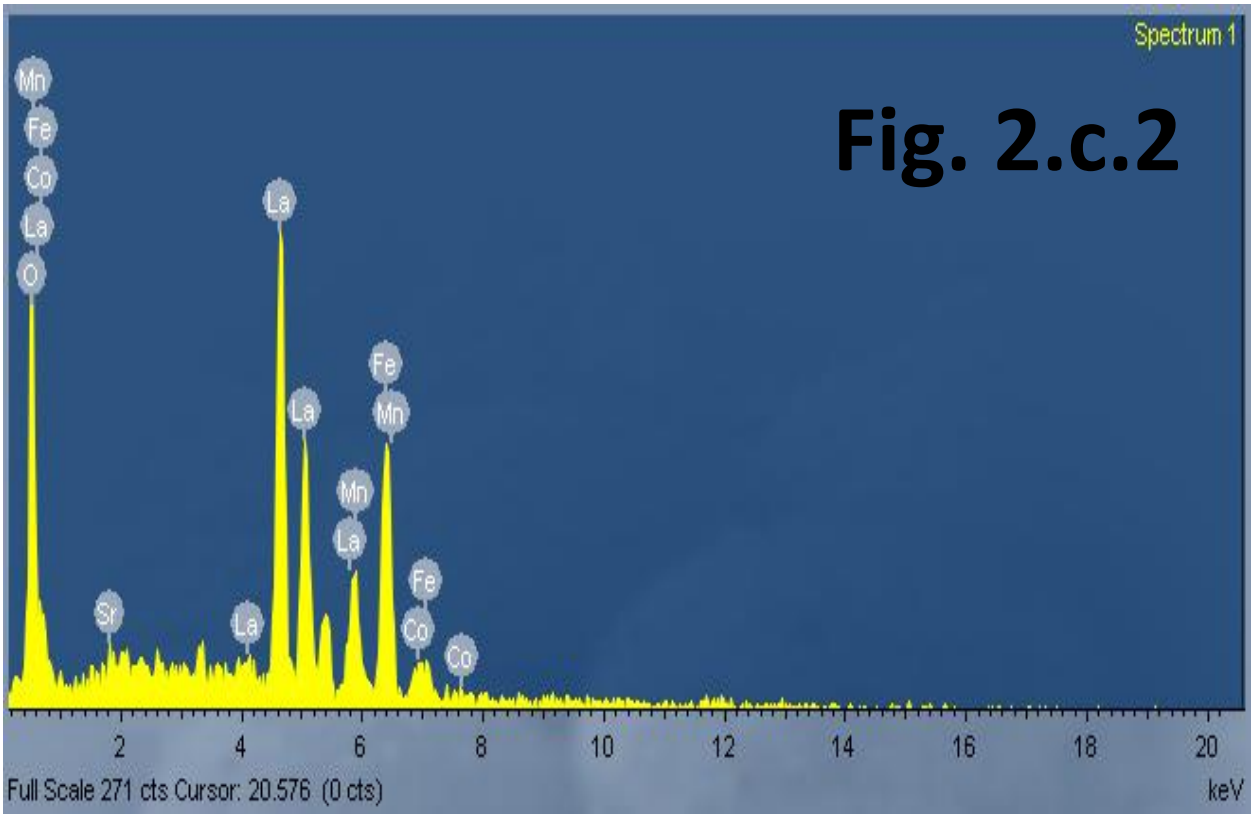


Fig.2.c. SEM Graph and EDAX for the composite composition 95LSMO:05CF
Fig.2.c.1 This represent the EDAX for the CF nanoparticle
Fig.2.c.2 This represent the EDAX for the LSMO nanoparticle

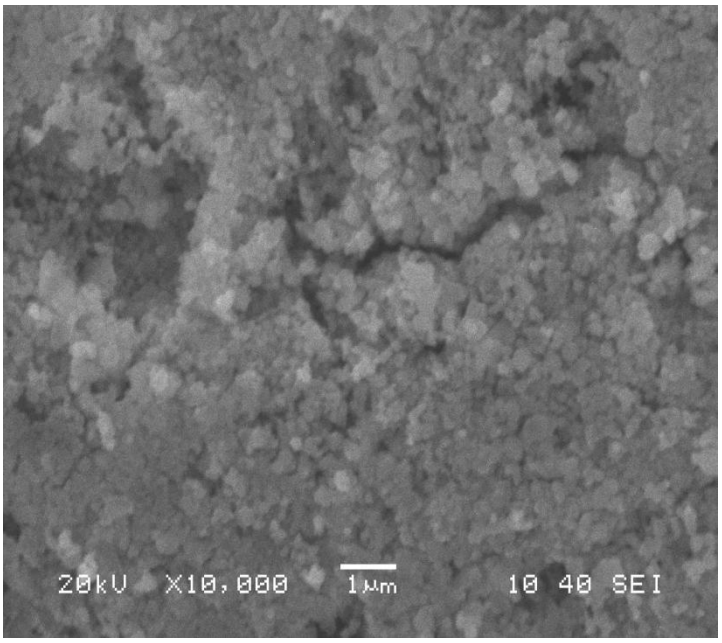
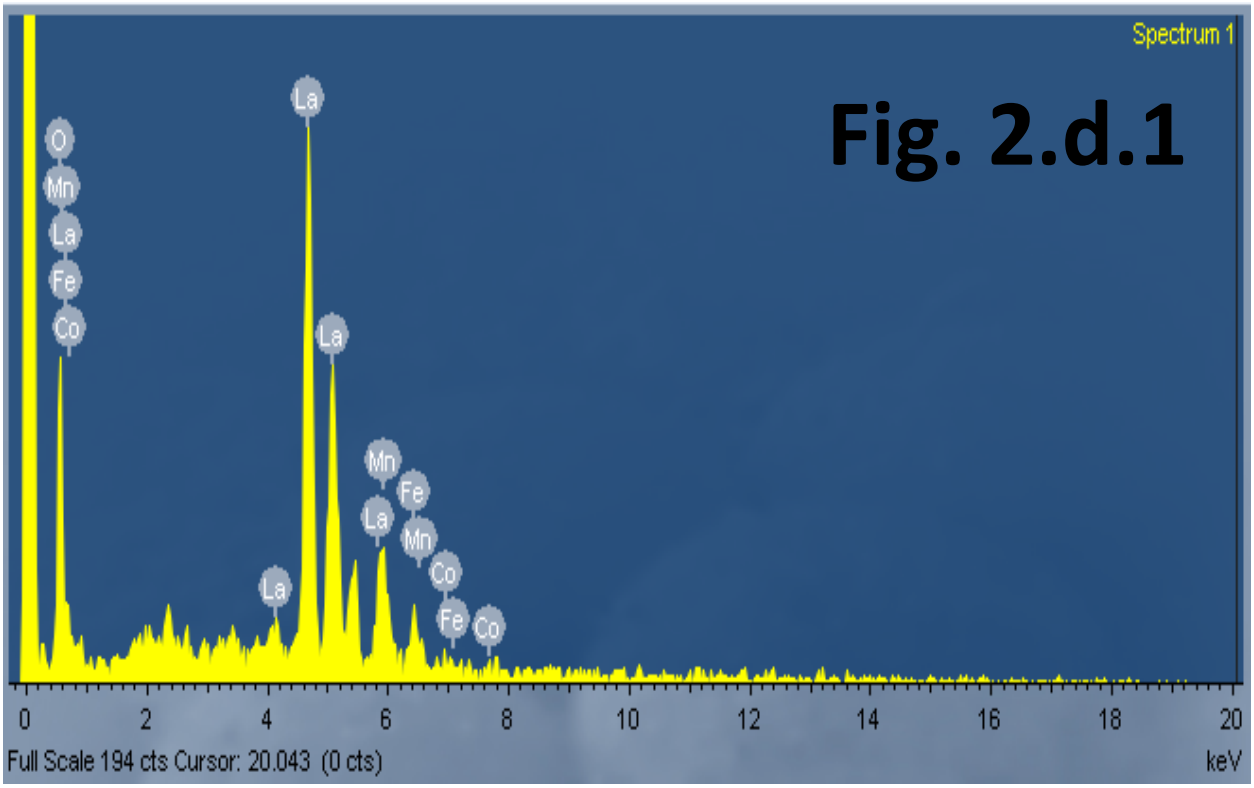


Fig. 2.d



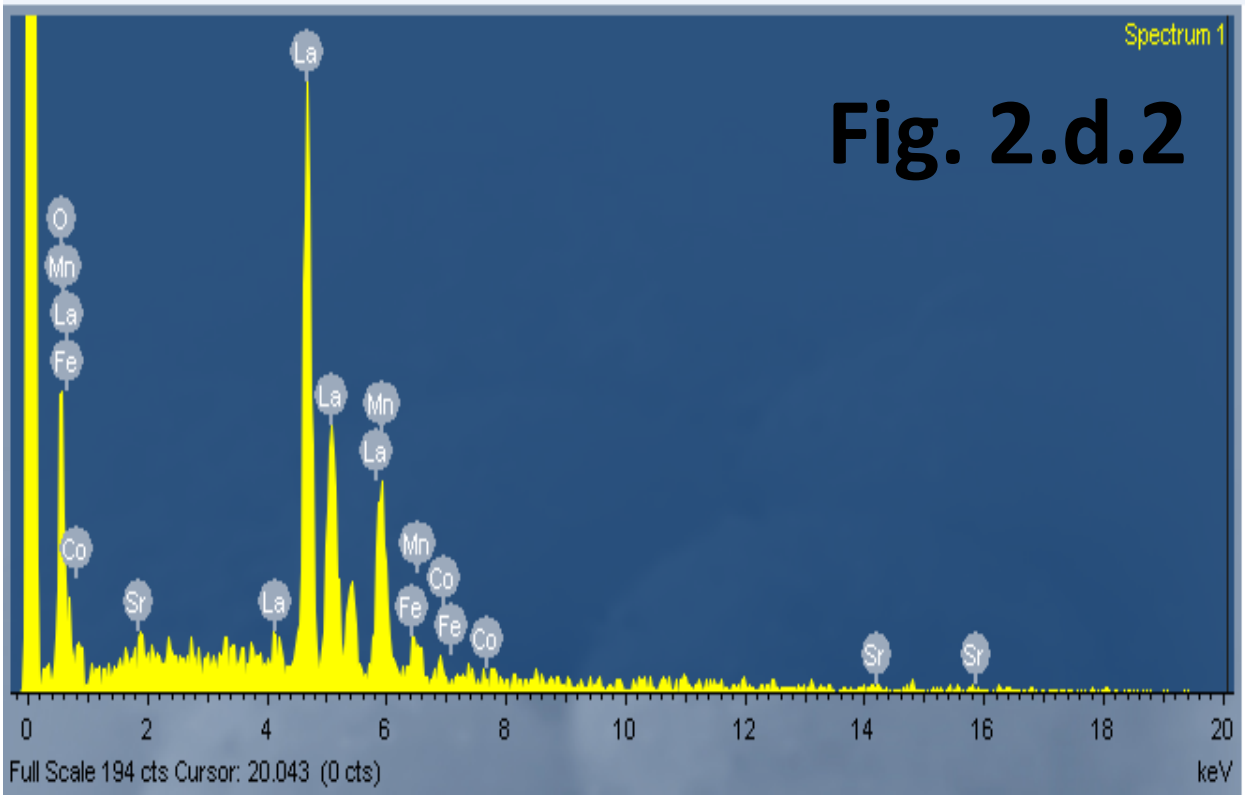


Fig.2.d. SEM Graph and EDAX for the composite composition 98LSMO:02CF

Fig.2.d.1 This represent the EDAX for the CF nanoparticle.

Fig.2.d.2 This represent the EDAX for the LSMO nanoparticle.

4.2.3 Density

The density of all sintered composites is given in Table 4.1. The density was at an average more than 85% in general was observed for all composites due to its morphology. Density increases with increase in CF content and highest density was obtained for 70 mol % CF addition.

LSMO: CF (mol %)	Dry weight (g)	Suspended weight (g)	Soaked weight (g)	Bulk Density[avg.] (g/cc)	Relative density in (%)
98 LSMO: 2CF					
Sample 1	0.6133	0.6156	0.6558		
Sample 2	0.5986	0.5274	0.631	5.4	85
Sample 3	0.6264	0.5514	0.669		
95 LSMO: 5CF					
Sample 1	0.4961	0.4229	0.4985		
Sample 2	0.5214	0.4578	0.5235	5.70	85
Sample 3	0.522	0.4496	0.5242		
90LSMO:10CF					
Sample 1	0.5511	0.4863	0.553		
Sample 2	0.6216	0.569	0.6235	7.41	86
Sample 3	0.6089	0.5398	0.611		
85LSMO:15CF					
Sample 1	0.5793	0.5045	0.581		
Sample 2	0.6216	0.5614	0.6285	7.87	87
Sample 3	0.6089	0.6016	0.6663		

Table 4.1 Density of pellets 98LSMO:02CF, 95LSMO:05CF, 90LSMO:10CF AND 85LSMO:15CF

4.2.4 Magnetization

The magnetization as a function of field at a room temperature for different composites (2 mol%, 5 mol%, 10mol% and 15 mol% CF content) are shown in Fig.3(a)-(d).This summarizes the magnetic data of the LSMO: CF composite. Certain microstructure of composite plays a very important role in the enhancement of magnetic properties.

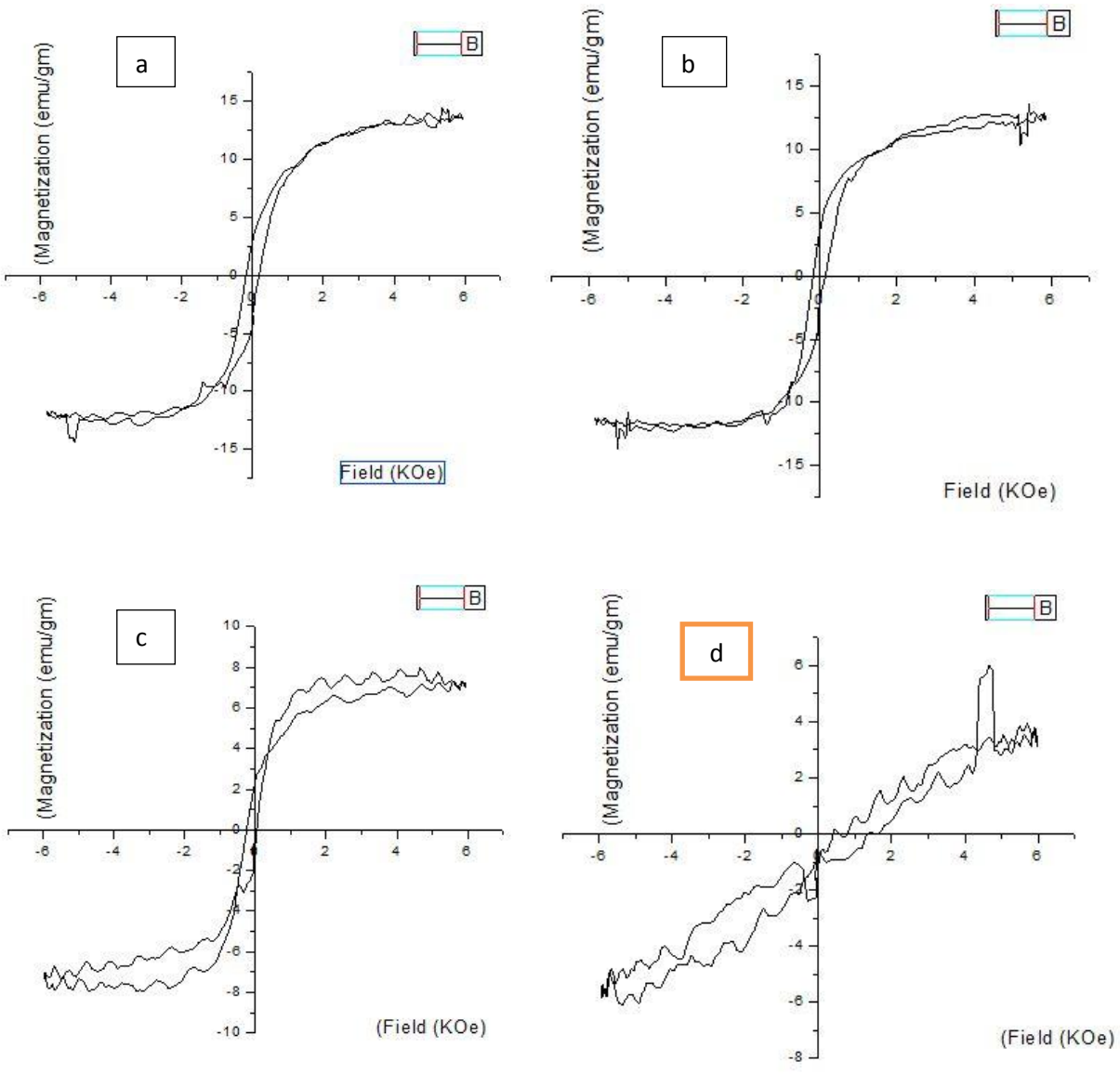


Fig.3: Room temperature M-H loop of microwave oven synthesized LSMO: CF composites having compositions (A)85LSMO:15CF (B)90LSMO:10CF (C)95LSMO05CF (D)98LSMO:02CF

Table 4.2: Magnetic data for the different compositions of LSMO: CF composites sintered at 1200^0C.

Sl No	Sample	Hc(Oe)	Ms(emu/gm.)	Mr(emu/gm.)
1	85:15	220	13	6.27
2	90:10	165	12	4.07
3	95:5	104	7	2.34
4	98:2	15	3	10.23

The saturation magnetization increases due to the increase of ferromagnetic order of CF, since coercivity is strongly depends on the microstructural property such as defects, strains and non-magnetic phases in the material. The 85 mole% LSMO: 15 mole% CF composite shows higher coercivity as well as higher saturation magnetization. This may be due to grain boundary pinning effect. Spin polarized intergrain pinning is due to magnetic disorder and magnetic coupling between LSMO and CF.

4.2.5 Summary

Nano grained composite of LSMO and CF were successfully prepared using microwave refluxing synthesis technique. In composite LSMO shows orthorhombic crystal structure whereas CF phase is cubic. The particle shape all composites show irregular shape. The average particle size distribution of all composite are found to be in the range between 200 nm to 300 nm. The 85 LSMO: 15 CF composites show higher saturation magnetization as well as higher corecivity.

Chapter – 5

Conclusions and future work

5.1 Conclusions

The significant findings of this work are:

1). The applied synthesis route,Kitchen microwave oven synthesis enable us to haveparticular type of microstructure and magnetic properties of the sample.

2). All samples show nearly spherical in shape as seen from SEM micrographs.

3). Particle sizes of LSMO grains arefound tobe smaller than CF grains.

4). Ms and Hc both are increased with increasing percentage of CF. This is due to the presence of insulating phase, which forces the motion of electrons to move (bend) through the manganite grains, thereby increasing significantly the contribution of grain boundary to the conduction process. Since corecivity is a microstructural property, 85LSMO: 15CF composite gives higher corecivity. This may be due to its unique morphology as compared to other composites.

5.2 Future work

The present work leaves a wide scope for future investigators to explore many other aspects like its structural stability property, magneto-electric effect at room temperature and high temperature, colossal magneto resistance (CMR) applications etc. The effect of internal strain on the magnetic property of this composite can also be studied along with the study of dielectric property in high temperature application.

Chapter- 6

References

6.1 REFERENCES

1).S. Asthana, A. K. Nigam and D. Bahadur, *Phys. Stat. Sol. B*, **243**, 1922–1928 (2006).

2). S. Jin, T. Tiefel, M. McCormack, R. Fastnacht, R. Ramesh, and L. Chen, *Sci*., **264**, 413 (1994).

3). C.N.R. Rao, *Mater. Today*, 9- 13,(2006).

4). R. Desfeux, S. Bailleul, A.D. Costa, W. Prellier and A.M. Haghiri-Gosnet, *Appl. Phys. Lett.*, **78**, 3681, (2001).

5). D. Shriver, P. Atkins, T. L. Overton, J. P Rourke, M. T Weller and F. A. Armstrong, "*Inorganic Chemistry*" W. H. Freeman, New York, (2006).

6). A.D. Andres, M. Garcıa-Hernandez and J.L. Martınez, *Phys. Rev. B*, **60**, 7328 (1999).

7). Z.F. Zi , Y.P. Sun, X.B. Zhu, C.Y. Hao, X. Luo, Z.R. Yang, J.M. Dai, W.H. Song, *Jour. Alloys Comp*., **477**, 414-419 (2009).

8). C.S. Xiong, F.F.Wei, Y.H. Xiong, L.J. Li, Z.M. Ren, X.C. Bao, Y. Zeng, Y.B. Pi, Y.P. Zhou, X.Wu and C.F. Zheng, *Jour. Alloys Comp*, **474** , 316-320 (2009).

9). C.H.Yan, Z.G.Xu, T.Zhu, Z.M.Wang, F.X.Cheng, Y.H.Huang and C.G.Liao, *Jour. Appl. Phys*., **87**, 5588 (2000).

10). S Das, A K Mukhopadhaya, S Datta and D Basu, *Mater. Sci*., **31**, 943–956 (2008).

11). R.David and D.Michael, *Jour.Organometallic Chem*., **384**, C57-C60 (1990).

12). B. B. Nayak, S. Vitta and D. Bahadur, *Maters. Sci. Engg. B*, **139**, 171–176 (2007).